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ARF-B215-12
(Summary Report)

STRESS DEPENDENT INTERACTIONS

BETWEEN CESIUM AND OTHER MATERIALS

Office of Naval Research
Power Branch
Washington 25, D. C.
Attention: Lt. Cdr. J. J. Connelly, Jr.

Contract Nonr 3441(00)
Modification No. 1

ARMOUR RESEARCH FOUNDATION
of
ILLINOIS INSTITUTE OF TECHNOLOGY
Technology Center
Chicago 16, Illinois

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ABSTRACT

Metals and alloys germane to thermionic energy converter usage have been screened for embrittlement by liquid cesium metal. The results of the screening evaluations are reported and techniques are described for more detailed studies of ceramics and susceptible metals.

Definite reductions in tensile ductility were observed for 302 stainless steel and molybdenum. Bend ductility was lowered in the cases of unalloyed titanium, titanium-6 aluminum -4 vanadium alloy, columbium, tantalum and silver-copper eutectic solder.

Some difficulty in reproducibly wetting samples was evident throughout the study. Slightly contaminating the cesium with oxygen (or water) was observed to increase cesium wettability markedly.

TABLE OF CONTENTS

LIST OF ILLUSTRATIONS

LIST OF TABLES

STRESS DEPENDENT INTERACTIONS BETWEEN CESIUM AND OTHER MATERIALS

I. INTRODUCTION

This report summarizes the results of studies during the period February 15, 1962 to February 15, 1963 on contract Nonr 3441(00), Modification No. 1. It summarizes work done at ARF under the direction of D. W. Levinson with the cognizance of Cdr. J. J. Connelly of ONR.

The study of embrittlement of metals, alloys, and ceramics by liquid cesium metal was undertaken to provide a fund of knowledge to prevent inadvertent failure of thermionic energy converters by an unfortunate choice of materials. The thermionic energy converters are operated under conditions of cleanliness which would encourage wetting of the internal and structural components of the diode by the cesium liquid and vapor. Wetting of the emitter and collector by the vapor is, in fact, required for the proper functioning of the diode. The concern which motivated the study can perhaps be better appreciated by consideration of the following sections of this report.

II. EMBRITTLEMENT BY LIQUID METALS

It has long been known that normally ductile metals can behave in a non-ductile fashion when wetted with certain liquid metals. An example of this is given in Figure 1, which shows the distinct difference in the behavior of 70-30 brass when wetted with mercury and subjected to the same mechanical abuse (a simple twist) as a dry specimen. The really brittle character of the fracture can be seen in Figure 2, in which the spiral fracture path of a brittle body which failed in torsion is shown in both the chalk (brittle) and the mercury-wetted brass (also brittle) samples.

In both cases, the brass is undergoing a premature failure at stresses and total plastic strains well below those values normally expected of the alloy. Thus one normally encounters a failure of the

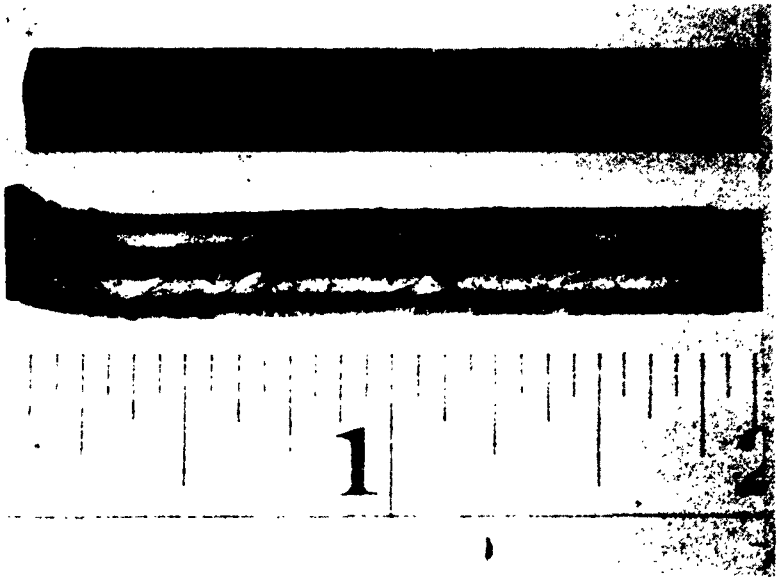

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Fig. 1

Premature Failure of 70-30 Brass Induced by Liquid Mercury

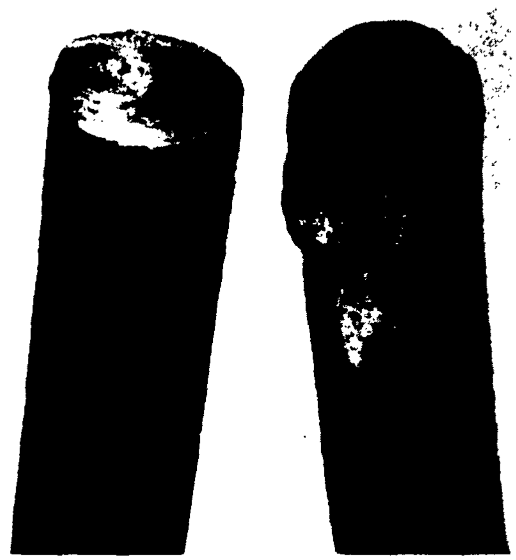

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Fig. 2

Similarity of Fractures of Chalk and 70-30 Brass, Wetted with Mercury in Torsion

type indicated upon the schematic sketch of Figure 3, varying in severity from measurable, as in (A) to catastrophic as in (B). Normally, the material has an ultimate strength σ_n and undergoes plastic strain ϵ_n prior to failure. These values are reduced to σ_A or σ_B and corresponding ϵ_A and ϵ_B by the presence of an embrittling liquid or vapor. Notice that it has been indicated that the stress-strain curve is unchanged, the manifestation of the embrittling effect simply appearing as a premature fracture.

In some cases such as high-strength aluminum alloys embrittled by mercury, failure occurs at stresses which are below the normal engineering yield stress. This implies totally brittle behavior (failure without plastic strain) unlike condition B shown on Figure 3, in which small but finite plastic strain ϵ_B is experienced. It is currently felt that completely brittle fracture is not likely and that failures below the so-called yield strength reflect simply the fact that less than 0.02% plastic strain (the yield strength value, by definition) is required to induce cracking in severe cases.

The mechanism of liquid metal embrittlement is not yet well understood. In many respects this embrittlement resembles stress-corrosion cracking, particularly with respect to the specific nature of the attack. Mercury catastrophically embrittles 70-30 brass (case B, Figure 3) but has no measurable effect on pure copper. Similarly, 18-8 stainless steel is "embrittled" in boiling aqueous $MgCl_2$ whereas other austenitic iron-base alloys are not. There exists (as with stress-corrosion cracking) a multitude of observations, some of which are in conflict. There are, however, some rather well-documented effects that appear to be general in character.

Temperature has a moderating effect upon liquid metal embrittlement. The work of Rozhanski et al.(1) indicates the normally observed behavior, which is shown in Figure 4. Polycrystalline aggregates behave, as a rule, in the same way, albeit at much smaller total strains. This behavior constitutes a transition from ductile (high temperature) to brittle (lower temperature) behavior. The range of temperature over which the change occurs is usually quite narrow, and thus a "transition temperature" is often mentioned for this phenomenon. It is, of course, of tremendous importance to realize this in the design of experiments (an exception has not

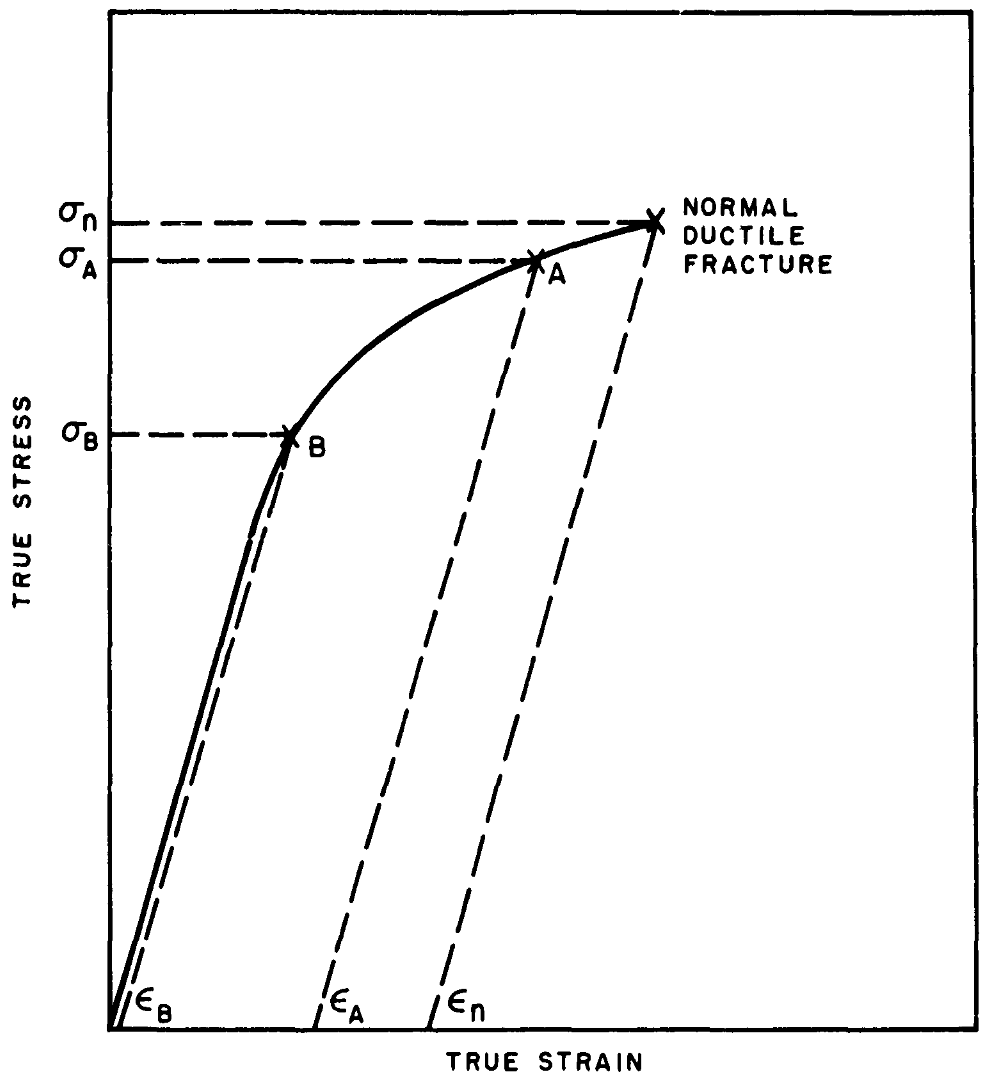

FIG. 3 - SCHEMATIC MODE OF EMBRITTLEMENT BY LIQUID METALS

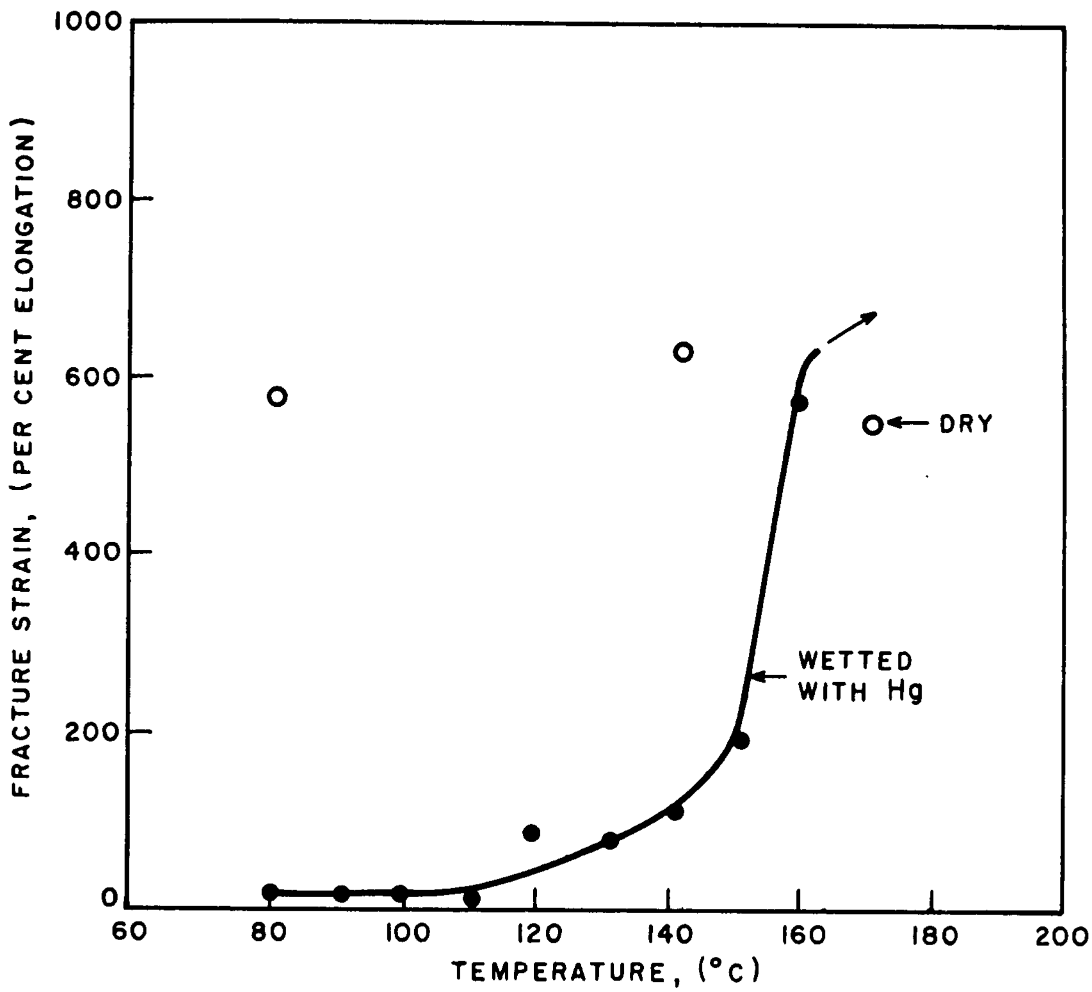

FIG. 4 - DUCTILE-BRITTLE TRANSITION IN MERCURY-WETTED ZINC MONOCRYSTALS

yet ever been found) since the susceptibility to this kind of cracking will be found at low temperatures (for the liquid) if it is to be found at all.

The embrittlement of zinc single crystals is an exception to the usual tendency for this phenomenon to influence only alloys of high flow stress--that is, strong materials.

Temperature has at least one second-order effect in that the propagation of cracks seems to require flow of liquid metal to the crack root to maintain the embrittlement. This is perhaps not true of extremely notch-sensitive materials, but would be expected to be true of most engineering alloys. The viscosity of a liquid metal varies with temperature (decreases*), and thus an increase in temperature should increase the initial rate of crack propagation. As the crack lengthens, frictional losses offset this and the crack velocity should become primarily inversely dependent upon crack length. There is evidence that this is so.[2, 3]

The principal effect of an increase in temperature to eliminate liquid metal embrittlement negates the notion that the phenomenon is one of very localized melting.

As might be anticipated in phenomena involving the rheological behavior of polycrystalline solids, the rate of straining is a parameter of some importance. Here again, there is probably a primary effect and a second-order effect. The primary effect is that of rate of strain, itself. This primary effect is seen to simulate the usual dependence of rupture stress on time. At the longer holding times (or smaller loading and hence smaller straining rates) the stress to produce fracture is lower than at higher strain rates. That is, plots of stress to rupture versus time have a negative slope. Because of the small difference in total strain at fracture, however, a test

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*Aluminum shows a viscosity increase with temperature up to about 1600°C which is due to oxidation. At 1600°C the oxide apparently dissolves in the Al, and a normal viscosity temperature relationship is resumed. Evidence gathered on this program shows that liquid Cs behaves this way if traces of O_2 or H_2O are present.

with relatively high strain and high strain rate, such as a small radius bend, is often apparently more severe than slow tension or static stress tests.

The secondary effect of high strain rate which can complicate efforts to study the effect of strain rate is again due to the inability of a molten metal film to follow the advancing crack. The impelling pressure will rarely exceed one atmosphere.

It is especially important to realize that delayed failure or static fatigue exists and is evident at stresses below those which will produce failure or large reduction in strain at fracture in a tensile test over the normal range of strain rates. Here, the behavior is especially analogous to stress-corrosion cracking. This, too, is important to the design of experiments.

The ductile-brittle transition mentioned earlier is also observed for polycrystalline aggregates, and the transition temperature is usually observed to be grain size dependent. In the case of the mercury/70-30 brass couple, a variation of grain size from 0.003 mm to approximately 0.1 mm produces an increase in transition temperature from 150° C to nearly 350° C.[3] That this is a complicated rather than simple effect can be appreciated by reference to Figure 5.

A secondary effect of grain size if that of increasing stress to fracture (wetted) as the grain size decreases.

The suspicion that this latter effect may simply mirror the requirement of a small but finite plastic strain to precede the generation and propagation of a crack is abetted by a data comparison such as Figure 6 allows. The yield strength is, as is well known and shown on Figure 6, grain size dependent. These data are for 70-30 Brass taken from the Metals Handbook.[4] The solid line on Figure 6 is data taken from a specific set of samples as quoted in Reference 3. The curves are strikingly parallel. The differences in the specific batches of material could easily account for the major portion of the separation between the curves. The significance of grain size is therefore not clear. However, at least in the case of 70-30 brass, the effect of yield strength is eminently clear. As the yield strength

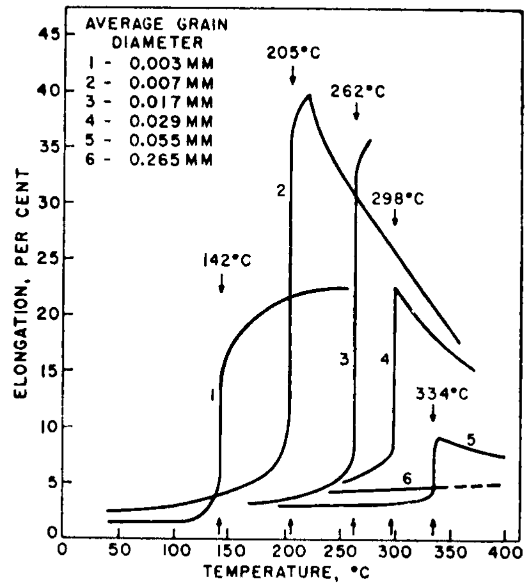

FIGURE 5 - EFFECT OF GRAIN SIZE ON THE BEHAVIOR OF 70-30 BRASS

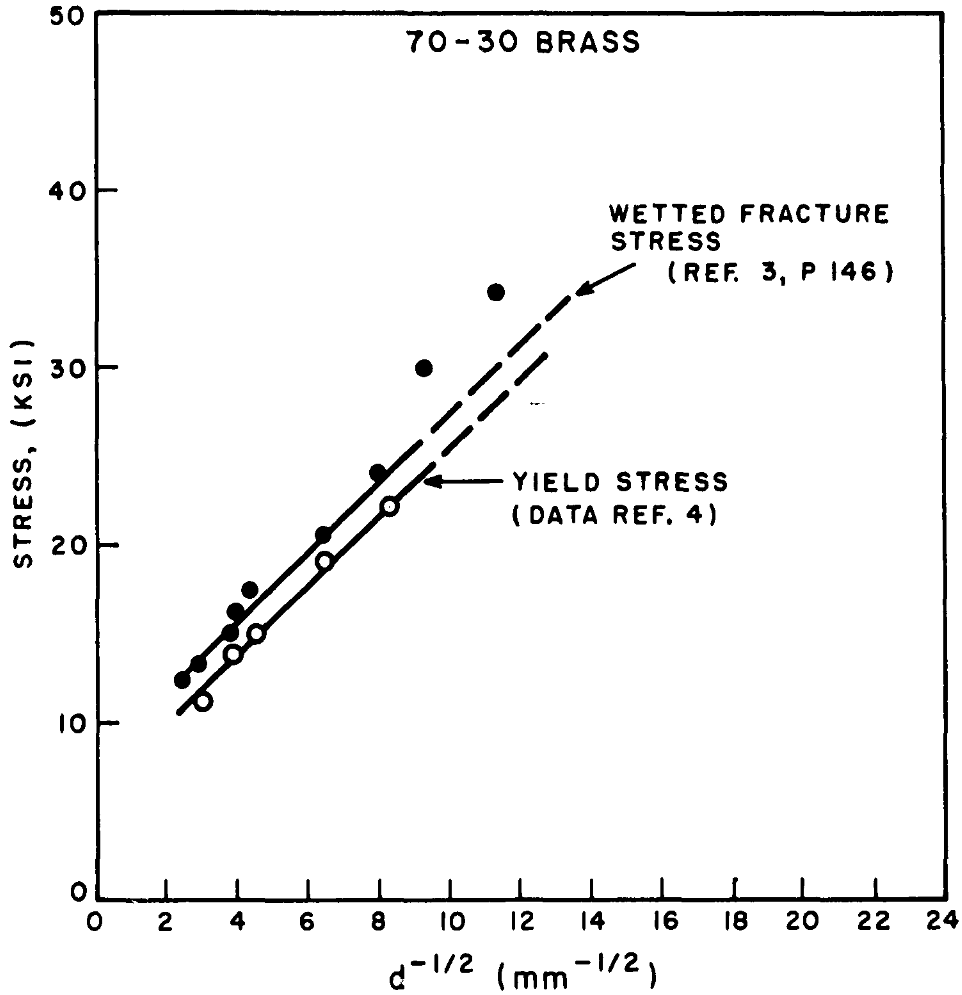

FIG. 6 - FLOW AND FRACTURE OF 70-30 BRASS

is increased by prestraining, the wetted fracture stress is accordingly increased to a value less than 1000 psi above it. These experiments were run on this program to elucidate this point and will be described in detail.

Metallographic observations indicate that the fracture is usually intergranular. Fracture paths have been observed to detour through elongated grains rather than around, and studies already cited (see Figure 4) of single crystal embrittlement indicate that the effect is not necessarily confined to grain boundaries. Where intergranular fractures are observed and liquid or vapor metal is present, this mechanism of liquid metal embrittlement is to be suspected. A typical fracture is shown in Figure 7. The locus of the main fracture is intergranular. Some transgranular satellite cracks are evident. This type of embrittlement has been frequently observed where no other obvious mode of attack (corrosion, dissolution, compound formation, or intergranular penetration) is evident, although the other forms of attack mentioned may also be present.

As a result of metallographic observations, one is led to conclude that the embrittling liquid somehow causes de-cohesion to occur, most often at grain boundaries, in the presence of tensile stress sufficient to cause some plastic flow.

It is clear that hardened alloys are more susceptible or, rather, more profoundly embrittled than the same alloys in softer tempers. In age-hardenable materials, for example, the severity is greatest just prior to, or at the hardness peak.[5] This, also, is important to the design of experiments, as will be seen later.

There have been no successful attempts to correlate the incidence of liquid metal embrittlement with crystal type or any aspects of constitution, composition, or phase diagram type. The phenomenon remains one of specific occurrence. A tabulation of sensitive systems is given by Rostoker,[3] and it is apparent that little relating to the effect of alkali metals on structural materials exists in the open literature. Sodium is known to embrittle aluminum and magnesium alloys, whereas lithium is known to attack steels but not aluminum and magnesium alloys. When one

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Fig. 7

Tensile Failure in Aluminum Alloy Coated with Mercury.

considers the spectrum of materials appropriate to the construction of thermionic energy converters and the entire absence of any systematic knowledge pertaining to the stress-dependent interactions between these and liquid or vapor cesium, a program of screening, to be reported in succeeding sections of this report, is of first-order importance.

This brief discussion of the liquid metal embrittlement phenomenon was presented for the sole purpose of acquainting the reader with the nature of this problem. Since non-stress dependent interactions between cesium and energy converter materials is under simultaneous study by Slivka,[6] the present work will limit itself entirely to studies in which the primary effect is that associated with the presence of stress, either applied or residual. For a review of phenomenology and a discussion of mechanism, the reader is encourage to consider the most recent reviews.[3,7]

III. EFFECTS DUE TO CESIUM

A fair quantity of information pertaining to effects of cesium on metallic and ceramic materials can now be found. A recent review of the effects of molten alkali metals on containment metals[8] has appeared but, aside from an acknowledgment of the fact that liquid metal embrittlement exists, no specific reference to the phenomenon is made.

Several effects other than the usual corrosion data are reported which are of direct interest to the thermionics program and of peripheral interest to this task. For example, pronounced intergranular attack by cesium of nickel was evident at 1832°F. There is little doubt that had the nickel been under stress, it would have failed. There is an important difference, however, between this type of intergranular attack which irreversibly embrittles the material and liquid metal "embrittlement." In the latter case the material is ductile if the "embrittling" medium is removed. This is an important distinction. In the former case one look at the microstructure enables the prediction that stress may have an adverse effect, especially if the liquid metal has wet the grain boundaries. In such an instance, the solid may well fall apart of its own accord. In the latter case, however, it is only after the fact that the effect of the presence of the liquid

is evident. As was mentioned before, there is no necessary unusual dissolution or intergranular penetration or film formation or other visible interaction. The mechanical behavior is simply different in the presence of the liquid than in its absence.

Thus, reports of intergranular attack of nickel[8] and the development of severe internal porosity in platinum, copper, and copper-gold alloys[9] are of primary importance to mechanical behavior of the affected materials, but are different from the interactions reported herein.

IV. EXPERIMENTAL STUDIES

The experimental studies reported herein are concerned with the susceptibility to liquid metal embrittlement of all materials pertinent to the thermionic energy converter. These include refractory metals, nickel-base alloys, stainless steels, titanium alloys, copper-base alloys, and high-alumina ceramics. A list of materials examined is presented in Table I. Studies during this contract period were essentially concerned with screening to determine susceptibility and were almost entirely concerned with molten cesium at temperatures near its melting temperature for reasons previously mentioned. Three experimental methods were employed. These were static bend studies for delayed failure, dynamic small-radius bend studies, and variable strain rate tensile studies. Details of the materials, methods, results, and conclusions are presented in the following sections of this report.

A. Materials

The structural materials listed in Table I were procured from a variety of local suppliers in the form of 1/4-inch diameter rod, 1/8-inch thick plate, and 1/16-inch thick sheet. In these forms the required samples were readily producible.

The refractory metals tungsten and molybdenum were of powder metallurgy origin and were typical of fine-grained wrought material. The grain size in both cases was of the order of 50μ average. The bend behavior indicated the tungsten sheet to be laminated. This is, unfortunately, typical of this material. It was not expected to be bend-ductile at room temperature. Both the tungsten and molybdenum were rather more ductile in tension than expected.

TABLE I

STRUCTURAL MATERIALS SURVEYED

Metal or Alloy	Nominal Composition wt%	Group
Unalloyed tungsten	99.9W	Refractory metals for hottest components--emitters and emitter supports.
Unalloyed tantalum	99.9Ta	
Unalloyed columbium	99.8Cb	
Unalloyed molybdenum	99.9Mo	
Kovar	Fe-28Ni-18Co	Nickel-base or nickel-rich alloys for metal ceramic seals, collector structures, internal supports.
Ni-Span C	Ni-40Fe-5.5Cr-2.5Ti	
Unalloyed nickel	99.5Ni	
Type 302 stainless steel	Fe-18Cr-8Ni (FCC)	Collector--typical alloyed stainless ferrous materials.
Type 430 stainless steel	Fe-14Cr (BCC)	
Titanium alloy 6Al-4V	Ti-6Al-4V	Base constituent important in reactive type metal-ceramic seals.
Titanium alloy B120 VCA	Ti-13V-11Cr-3Al	
70/30 Brass	Cu-30Zn	Typical sealing solder and reference material.
Copper-silver eutectic	Ag-28Cu	

The tantalum and columbium were fine-grained wrought material comparable in ductility to the best these metals have to offer, a testimony to their freedom from interstitial contaminants. At the time of initiation of these studies high-strength alloys of these metals were not readily available. It is not likely that refractory metal base alloys will find immediate application in diodes and the screening of high-strength alloys (desirable for reasons discussed previously) is in this case not of pressing urgency. They are to be systematically procured, however.

The other metals were received from suppliers in typical "mill annealed" form which for the 70-30 brass, is fully annealed, and for the stainless steels and titanium alloys is a moderate strength condition. All possessed microstructures typical of these materials.

The cesium was procured from Mine Safety Appliance Research Corporation, Pittsburgh, Pennsylvania, in a single 1 lb batch sealed in a hermetic stainless steel container. A chemical analysis of the material is given in Table II. Total impurity content of this material is stated to be 200 ppm maximum--not, however, counting oxygen, which was not specified. For reasons to be mentioned, the oxygen content was suspected to be quite low. The design of the container, furnished by the supplier, was such that the metal could readily be melted in the sealed container and transferred with ease to storage containers in a glove box which could be purged by evacuation (always to below 1 micron and, for some experiments, to 0.01 micron) and refilled with either tank purity or purified inert gas. Normally, argon gas of tank purity was quite adequate and, in most studies, desirable. The very slight water contamination markedly improved the wetting tendency of the cesium.

B. Experiments and Methods

1. Wetting Studies

The first requirement of experimental work in the field of liquid metal embrittlement is the achievement and maintenance of good wetting. There are several quantitative methods of measuring wetting, the sessile drop method being the most well known. This requires a configuration which is not

TABLE II

ANALYSIS OF CESIUM METAL

Element	Amount (ppm)
Cs	major
Fe	< 5
B	< 5
Co	< 5
Cd	--
Mn	< 1
Al	< 5
Mg	< 1
Sn	< 5
Cu	< 5
Pb	<10
Cr	< 5
Si	<25
Ti	< 5
Ni	< 5
Mo	< 5
V	< 5
Be	< 1
Ag	< 1
Ba	< 3
Sr	< 1
Ca	2
Na	40
K	< 5
Rb	75

a practical geometry for mechanical testing and, accordingly, wetting must be assessed by a rather qualitative judgment of the maintenance of a stable liquid metal film.

It is often found, too, that wetting can be transient. This is to say that a particular couple may readily wet and, some time later, because of oxidation of the solid metal or contamination by atmospheric content, or by dissolution from or into the liquid, suddenly de-wet. This is normally very obvious, the liquid metal balling up to destroy the continuity of the initial film.

It was mandatory for this program that techniques be worked out to insure good wetting initially and the maintenance of wetting at least for the lifetime of the test. This was as long as 100 hours in some of the static fatigue studies, though usually one-half hour was sufficient. As might be expected, cesium did not readily wet any of the materials, even after a cleaning procedure consisting of a hot aqueous detergent (scrub) followed by hot water and alcohol rinsing and immediate transfer to the dry box.

Various kinds of mechanical assistance were employed with varying success ranging from an ultrasonic soldering iron to scrubbing the immersed sample surface with stainless steel wool.

For these studies the procedure employed was to evacuate the dry box to at least 5×10^{-4} mm using a portable vacuum system (6-inch oil diffusion pump) and a liquid nitrogen cold trap. If the dry box was closed properly, this pressure could usually be achieved in 30 minutes. Pressure was measured with an ion gage just outside the chamber wall between the dry box and the cold trap. Argon was then admitted through a hot titanium trap and the system brought to just over 1 atmosphere. It was then possible to work through the wall in the gloves.

The measured volume of the chamber with the gloves extended was 268 liters. A set of experiments was run in which, substantially, the procedure above was followed except that systematic amounts of water vapor were introduced by dehydrating weighed quantities of $CuSO_4 \cdot 5H_2O$ on

a watchglass heated electrically by a small Calrod heater. Amounts of water up to 0.1 gram were added yielding a set of water partial pressures up to approximately 350 microns. These values were calculated assuming water, at this pressure and dispersed in an inert gas, to behave according to the ideal gas law. At these pressures this is a good assumption.[10] Upon the conclusion of these experiments, the use of gas purification was discontinued, and the bulk of the studies reported involved the use of tank argon. The dew point of the gas used was -70°F, representing partial water pressures high enough to facilitate wetting. The introduction of controlled amounts of water into the system is the result of analogous behavior observed with "self-fluxing" brazing alloys containing lithium. These remarkable alloys readily wet metals like stainless steel in air but will not do so in atmospheres essentially free of water vapor. This was a surprising result when first discovered, but is now well understood[11] and is the result of the formation of a low-melting hydroxide of lithium. In the present case it remains unclear whether this improvement in wettability of the cesium on all of the materials studied is due to dissolved oxygen or to the formation of a hydroxide, as in the case of the brazing alloys. It is eminently clear that wettability is enormously improved.

It was desired to find a method for maintaining the wetted cesium film, at least long enough to permit transfer from the dry box (Figure 8) to the tensile testing fixture (Figure 9). If necessary, the bending data and static fatigue could be gathered within the protective atmosphere, and the tensile capsule could be loaded in the dry box, but at considerable inconvenience. It was reasoned that inert organic liquids might provide adequate protection. Refined kerosene, Nujol (highly refined mineral oil), Xylene, white paraffin oil, silicone DC 200 oil, and refined petrolatum were selected. All but the Xylene and silicone are straight aliphatic hydrocarbons. Xylene and the silicone oil did not adequately protect the coated samples. The cesium oxidized rapidly and completely. There may have been a reaction between the cesium and either or both of these liquids. Kerosene did not appear to react, nor was it adequately protective. Again, the cesium would oxidize fully within 15 minutes at 20°C, a temperature at which the

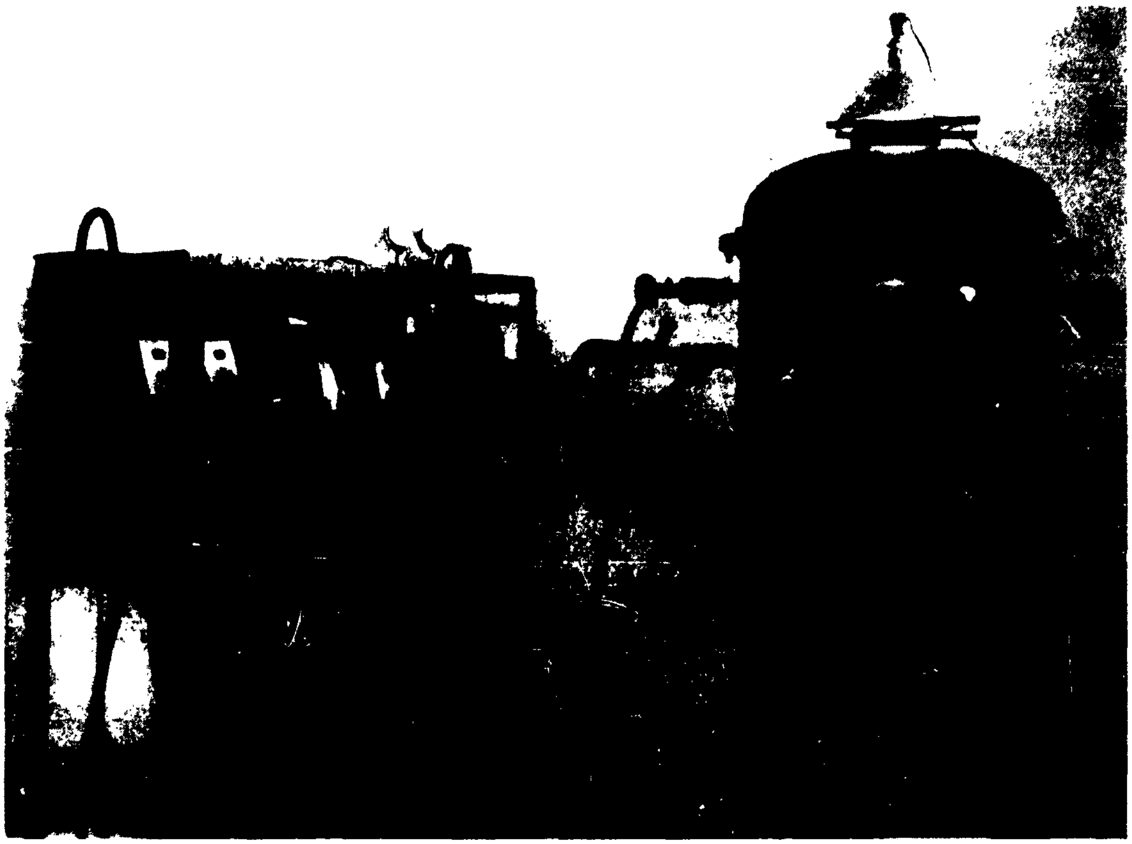

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Fig. 8
Vacuum Dry Box Used For Specimen Handling

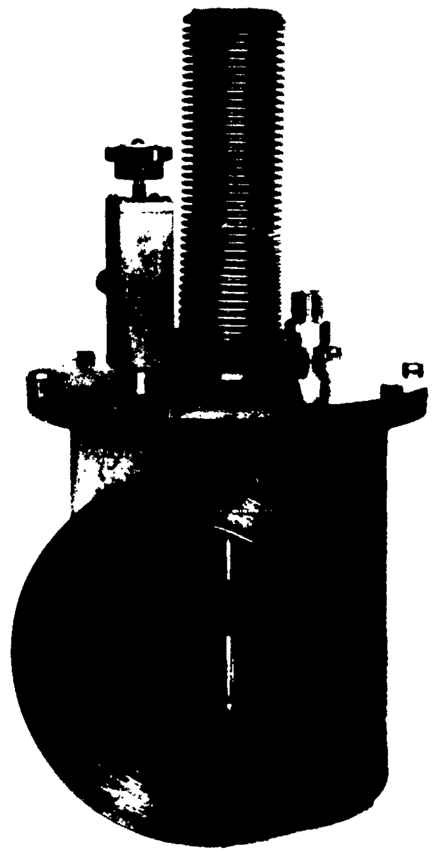

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Fig. 9
Tensile Test Chamber

cesium was a solid. Both the mineral oil and the paraffin oil were more protective (adequately so) as long as the cesium was frozen and the samples were fully immersed. Although the cesium did not de-wet, it did become obviously oxidized if the sample was removed from the oil bath and allowed to drip. The thin oil film was not adequately protective.

A heavy film of refined petrolatum was found to provide adequate protection to prevent oxidation and de-wetting of the cesium even when the cesium film was molten. As a result, samples for study could be wetted, coated, and transferred readily to test fixtures; tests were carried out either in the protective chambers, or, in the case of the dynamic bend studies, which are quite rapid, in air.

In order to avoid the possibility that the samples would literally run out of cesium during the tensile studies, collars of low-density 18-8 stainless fiber metal were fabricated and used both as abrasion pads to facilitate wetting and as reservoirs during the test. A typical petrolatum and cesium coated tensile sample is shown in Figure 10 with the reservoir collar evident.

These samples were threaded 1/4-20 on the ends and had a 1/8 inch gage diameter and a 1 1/4 inch gage length. They were usually stored at or below 20° C (cesium solid) prior to actual testing. The cesium was melted by radiation from a 500-watt reflector lamp after the sample was attached within the test capsule and ready for stressing. The petrolatum served as rather useful thermal insulation, allowing the sample and cesium to remain cold enough to keep the cesium frozen during loading of the test fixtures. The petrolatum was adequately transparent to allow viewing of the cesium film and thus ensure that dewetting had not occurred.

2. Static Bend Studies

The susceptibility to static fatigue was determined by fabricating specimens from 1/16 inch sheet. The samples were 1/4 inch wide and 2 3/4 inches long. These were centrally loaded as simple beams (end supported) in a fixture shown in Figure 11. Stresses were calculated from the simple beam formulas, and deflections were measured with a dial

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Fig. 10
Tensile Test Samples

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Fig. 11

Static Bend Stress Fixture

gage. It was later found more convenient to handle samples individually, and the fixture was sliced up into invididual sample holders. Several runs were made at stress levels of 0.7 and 0.9 of the measured yield strength of the material and one set of determinations at stresses, in each case, 10% in excess of the yield. For these determinations petrolatum protection within the dry box was the most satisfactory procedure, and 100-hour runs were successfully completed. During these runs, the dry box atmosphere was maintained at 30° C.

3. Dynamic Bend Studies

These studies were carried out on strip samples identical to those used for static bending except in the case of composite samples for the brazing alloys. Here a sample of Kovar and braze alloy was fabricated by wetting a prepared cavity in a strip made from 1/8-inch plate stock with the brazing alloy, allowing the composite to cool, and then machining a bend sample as shown in Figure 12. This geometry permits the brazing alloy to be placed either in uniaxial tension or in tension from bending. This latter mode of stressing was used for the data reported herein.

In all, three different sets of dynamic bend data were obtained. These were bends to 90° around 2t and 3t radii (t is sample thickness), to 180° around 2t and 3t, and a series of "sharp" bends, to 90° around a pin 1/16 inch in diameter. A standard Di-Acro bend tester was used for these studies. One set of sharp bends was made over a ground plate in a vise.

Here, again, the most successful procedure involved the use of a protective coating of petrolatum. Samples, again, were wetted, coated, frozen in the dry box, and then warmed by radiation from a lamp to 30° C and tested.

4. Tensile Tests

Tensile tests were carried out in the chamber previously shown which could be independently filled with argon or, for one set of runs at high strain rate, out in the open. In all cases petrolatum-covered samples

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Fig. 12
Composite Braze Alloy Sample

were employed, and all testing was performed on an Instron machine with full autographic load-extension recording. The strain rates employed were 0.01 in./min, 0.10 in./min, and 0.5 in./min. Unwetted blank samples were used at the lowest and highest strain rates. In all cases full autographic stress-strain records were kept, the details of which will be discussed presently.

5. Other Experiments

Several sets of control experiments were performed with a familiar and susceptible system to check on the effectiveness of the tests anticipated and to clarify certain aspects of the embrittlement phenomenon about which there is controversy. All of these experiments used 70-30 brass samples and mercury as the embrittling liquid. The mercury was applied from an aqueous nitrate solution which plated mercury on the sample surface by electrochemical displacement. For elucidation of the results of fracture in different types of stress fields, tension, torsion, and bend studies of a very prototypic nature were conducted.

The tensile studies involved a comparative series, in which samples were strained to wetted fracture after dry prestraining. In this way the yield stress of the brass could be varied from 20,000 psi to over 40,000 psi without changing grain size, and the importance of yield strength, per se, could be examined.

Similarly, the onset of brittle fracture in a torsion test should appear in a very particular way (recall Figure 2). Future delayed failure studies can and will be run in this way, as will be shown in the final sections of this report.

One additional long-term static exposure experiment was begun based upon obvious discolorations found in some of the experimental samples. The studies are under way at this writing, and, unfortunately, no results are yet available.

The study of the susceptibility of materials to cesium "embrittlement" is rendered doubly difficult by the fact that the ceramics are brittle in tension initially. The question is really whether or not cracking will occur at lower stress in the presence of cesium rather than in its absence.

One major source of difficulty is in the achievement of axial loading. Bending stresses arising from alignment difficulties lead to erratic premature failure. Typically, one encounters large scatter in such data. The influence of a surface-active medium is thus assessable only in a statistical way unless its effect is catastrophic. Both brittle rings and θ specimens have been used with some success. Both, however, have drawbacks. The ring compression test has the great bulk of the working tension in a very small volume of material, and the θ specimen is difficult to make.

In principle, bending of brittle beams is able to produce tensile data if the beams are properly perforated. Bortz[12] has studied the influence of specific hole geometry on the purity of tension produced in the gage length and has established a feasible geometry. This study was based upon photoelastic analysis of beams as a function of loading and hole or slot geometries. An offset longitudinal slot analogous to a tied beam with pure tension in the tie rod proved to be the most satisfactory sample geometry (the sample is bent with four-point loading). The development of this geometry is shown in Figure 13.

Fabrication of specimens to this geometry has been achieved by "soft" firing the commercial Al_2O_3 (to date with Wesgo Al-995) to a blank, using a resin binder. Samples are machined, as is a loose insert between the body and tie rod portions of the sample. The samples are then fully fired. To date, tests of the reproducibility of the unwetted strength only have been performed.

V. RESULTS AND DISCUSSION

A. Wetting Studies

The principal results of the study of the wettability of metals, alloys, and ceramics by cesium are the following. Cesium will readily wet clean metal surfaces, provided that a small quantity of oxygen, water vapor, or both are present in the ambient atmosphere. It has not as yet been ascertained which constituent is of critical importance, nor has it been determined whether either is needed if the metal surface is really clean--that is, free of all traces of surface contamination. The important finding in this case is

2 Inch Transition Radius

3 Inch Transition Radius

Fig. 13
Photoelastic Beams

that the improved wettability afforded by the deliberate but slight contamination of the cesium enables cesium metal films to wet and remain on surface for periods long enough to determine its effect upon mechanical properties. During these studies it was observed that the cesium, which, when freshly drawn from its container, is brilliantly silver white in color, becomes slightly darker in color when contaminated by traces of oxygen or when allowed to react with traces of water vapor. The slightly discolored metal readily wet all of reasonably clean (washed and degreased) metal surfaces with which it was brought into contact.

It was observed that this can be carried too far. If the cesium is deliberately contaminated to the degree required for any solid oxide to appear, wetting is fully inhibited. This requires approximately 2.5w/o oxygen. Wetting may be inhibited at much lower oxygen contents, but this was not determined. This oxygen would be soluble in the molten cesium as shown in Figure 14.

The maintenance of a stable, wetted molten film on the surfaces of experimental samples required protection from oxidation. The unpurified tank argon in which wetting was found to occur readily would not maintain wetting for more than about 2 hours at 30° C. In gettered gas this difficulty was not encountered. As was mentioned, a heavy petrolatum film was found to provide adequate oxidation protection to inhibit de-wetting for periods of hours in open air at 30° C and for much longer periods in either tank or purified argon. The results of studies of various coating materials are summarized in Table III. Not only are the heavier molecular weight hydrocarbons adequate diffusion barriers to gases of the atmosphere, but they do not themselves produce dewetting.

B. Dynamic Bend Studies

The behavior of metals and alloys during dynamic bending, either guided or free, when embrittled by a liquid metal is typified by the results shown in Figure 15. Three conditions are shown. If a sample is wet before bending (in the figure 70-30 brass samples wetted by mercury are depicted), only slight bending deformation is tolerated before very definite cracking occurs. In the figure the mercury is removed by distillation

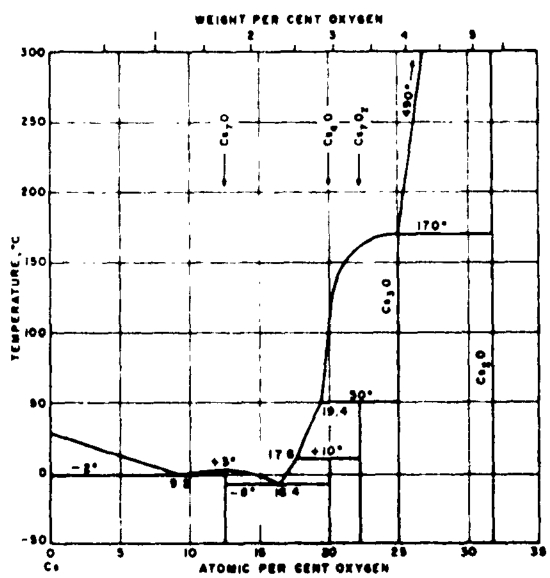
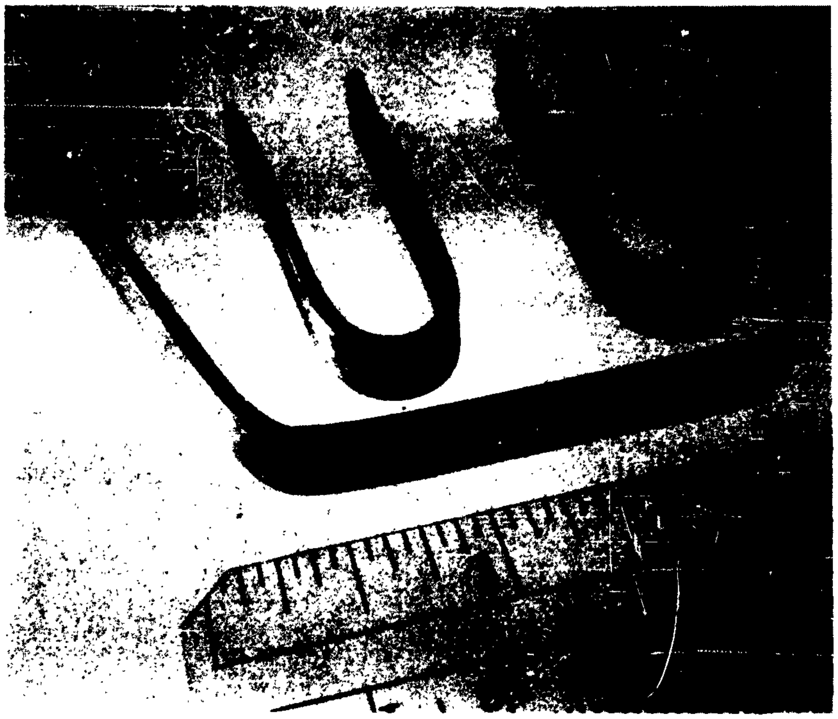


Fig. 14

The System Cesium-Oxygen



Neg. No. 24590

Fig. 15
Brass Bend Test Samples

TABLE III

RESULTS OF WETTING STUDIES OF COATED METALS AND ALLOYS

Coating Material	Cesium Solid (T < 28° C)	Cesium Molten (T = < 30° C)
Xylene	Metal oxidizes rapidly	Solid oxide appears and metal de-wets
Kerosene	Slow but ruinous oxidation	Rapidly oxidizes and de-wets*
Nujol	Slow oxidation**	Moderate oxidation rate (de-wets)
Parafin oil	Slow oxidation**	Moderate oxidation (de-wets)
Silicone oil	--	Poor protection offered (*?)
Silicone grease	Apparently inert	Apparently inert for short times
Petrolatum	Apparently inert	Apparently inert

* Apparent reaction (bubble formation) between cesium and the liquid.

** OK. for long storage provided samples are fully immersed.

to reveal the cracks clearly. If on the other hand, the sample is not wetted, the ductility characteristic of the alloy allows the full 180° 3t bend to be achieved easily. Wetting the sample after bending did not (as shown) produce any delayed failure in the several hours which elapsed, owing to the residual stress present in the sample. A similar bend sample tolerated approximately 25, 000 psi for over 100 hours (test was terminated) without a sign of cracking. Wetting prior to bending produced severe cracking beginning at about 19, 000 psi. The implication is that plastic flow must occur while the sample is in contact with the embrittling liquid in order for cracking to occur. This result led to additional studies of tensile behavior which will be discussed presently. The system mercury/70-30 brass is typical of behavior at point B on Figure 3, as will be shown.

The alloys of interest to this program were screened through three bend radii and the results are presented in Table IV. The increasingly smaller bend radii represent what has previously been observed to be a test of increasing severity. As can be appreciated from the data of Table IV, the only materials clearly indicated to be embrittled in the annealed condition are Ti-6Al-4V alloy, unalloyed titanium, and the BT solder. Molybdenum, tungsten, and (surprisingly) Ni-Span C alloy lacked ductility adequate for the dry versus cesium-wetted comparisons made. The titanium alloy B120 VCA was affected much less severely than the other two studied. The appearance of dry and wet Ti-6Al-4V samples is shown in Figure 16. Definite fracture is evident in the wetted sample on the right, whereas the dry sample shows a few satellite cracks only. The samples shown are sharp bends.

It is interesting and probably significant that the body-centered cubic (β) titanium alloy (B120 VCA) was much less severely attacked than either the $\alpha + \beta$ (Ti-6Al-4V) or than the all hexagonal-close-packed α (unalloyed) material. It is unusual to find a pure metal (unalloyed Ti) embrittled. It is, however, not unusual to find a distinct difference in behavior as a function either of composition or of constitution.

The significance of the moderate embrittlement of titanium can perhaps best be appreciated by recalling that many metal-ceramic seals are made using "activated" alloys or a titanium hydride layer on the ceramic which

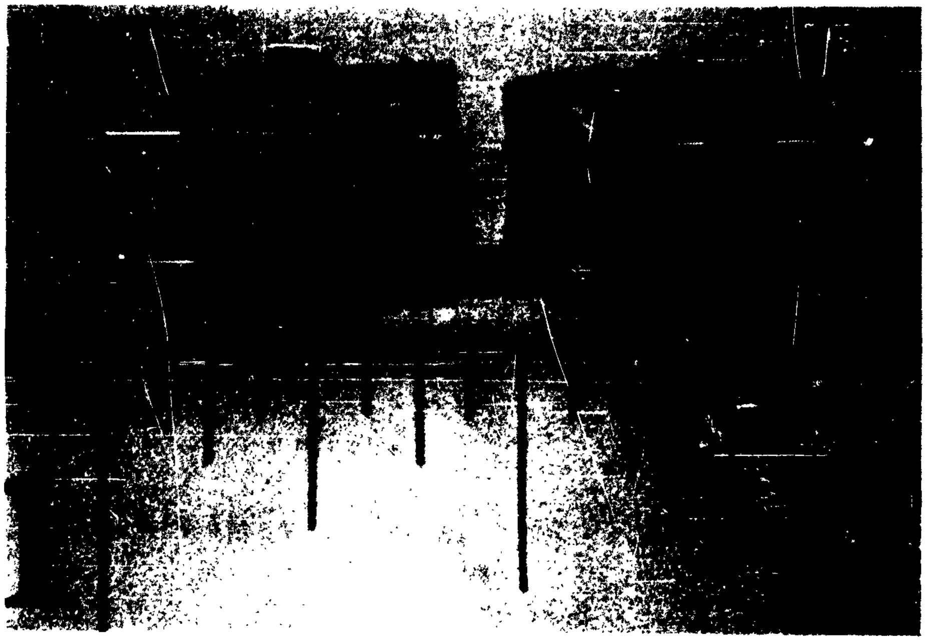


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Fig. 16
Ti-6Al-4V Bend Test Samples

is reduced during the brazing operation. Titanium has been found to segregate (see Figure 17) to both metal-braze and braze-ceramic interfaces, a position in which it could lead to cesium cracking of this type of seal. Figure 17 shows the distribution of silver (uniform) and titanium (strongly segregated) as determined by the electron microprobe. A diffusion gradient in titanium is evident at the braze-Kovar interface. There is no question that unless care in the design of such joints is taken, the interfaces may be stressed such that tensile components are present.

The presence of satellite cracks in only the wetted sharp bends of columbium and tantalum indicates that these materials may be embrittled to a degree by cesium also. Subsequent studies of Ta and Cb will therefore involve increasing the yield strength by cold working.

In all cases the results reported in Table IV are the predominant behavior observed in at least three separate determinations with identical specimens.

C. Static Bend Studies

A 100-hour wetted static bend run was computed to 0.7, 0.9, and 1.1 times the deflection shown to produce yielding in the simple beam samples used. A second run which was to have run for 1000 hours was terminated when a leak caused de-wetting of all of the samples.

In the run which completed 100 hours, no cracking was observed in any case. In all cases some staining or corrosion of the surface was evident though this may have resulted from corrosion by the highly alkaline residue resulting during the removal of the cesium. The samples were all essentially unattacked, no intergranular penetration or excessive dissolution could be found. An altered surface layer was found in the titanium 6Al-4V alloy, giving the appearance of a diffusion layer identical to that found on the dynamic bend samples.

These negative results are very encouraging, and the study will, of course, be repeated for materials in the higher strength configuration. There is apparently insufficient plastic flow at 30° C (as was

Ti

Neg. No. MP 43

Ag

Neg. No. MP 45

Fig. 17

Elemental Distribution in Kovar-Alumina Brazed Joints

TABLE IV

DYNAMIC BEND DATA

Material	3t Bend (180°)		2t Bend (180°)		Sharp Bend (180°)	
	Dry	Cs coated	Dry	Cs coated	Dry	Cs coated
B120 VCA	OK	OK	OK	OK	OK	Many small cracks +
Ti-6Al-4V	OK	OK	OK	OK	OK[+]	Fracture
Unalloyed Ti	OK	OK	OK	OK	OK	Fracture
70-30 Brass	OK	OK	OK	OK	OK[+]	OK[+]
BT Braze*	--	-	-	--	OK[+]	Fracture
Easy Flo 45*	--	--	-	--	OK[+]	OK[+]
Zircaloy-2	--	--	--	-	Fracture	Fracture
Mo	OK	OK	Fracture	Fracture	Fracture	Fracture
Cb	OK	OK	OK	OK	OK	OK[+]
Ta	OK	OK	OK	OK	OK	OK[+]
W	Fracture	Fracture	Fracture	Fracture	Fracture	Fracture
Ni	OK	OK	OK	OK	OK	OK
Ni-Span-C	OK	OK	--	Fracture	Fracture	Fracture
Kovar					OK	OK
430 Stainless	OK	OK	OK	OK	OK	OK
320 Stainless	OK	OK	OK	OK	OK	OK

* Laminate samples

\+ Some satellite cracks evident at sample edges

previously implied but not shown to be necessary) and, while stress relaxation is anticipated to some degree, it would be expected to alleviate rather than aggravate the tendency to delayed failure.

The determination of the role of creep in the fracture induced or propagated by a liquid metal still requires much elucidation. If time permits, attention will be devoted to this point during the forthcoming study period.

It is significant that a simple beam sample of 70/30 brass wet with mercury, after being stressed past the yield, failed in 49 hours. Exposure to aqueous mercuric nitrate solution is still regarded as a very sensitive test for residual stress in brass. This solution immediately plates mercury over the sample surface. Plotting published[4] creep rate data for annealed 70/30 brass versus reciprocal absolute temperature and extrapolating to 30° C indicates a strain rate of 2×10^{-4} per cent per hour, or a total strain of the order of 1.0×10^{-2} per cent to fracture at 20,000 psi. This is in reasonable agreement with the observations in tension tests that 70/30 brass wet with mercury fails just past the 0.02 per cent offset yield. The measured yield strength of this material is approximately 18 ksi.

D. Tension Testing

Tension test results tabulated here are preliminary and are the results of, at most, duplicate tests. It is to be expected therefore that some scatter and inconsistency may result which subsequent study will eliminate. The tensile test results are summarized in Tables V, VI, and VII.

Several aspects of these data are especially worthy of comment. Firstly, the tensile studies do not appear to give indications consistent with the bend results. Whereas dynamic bend studies showed definite (though in no case catastrophic) embrittlement of titanium and Ti-6Al-4V alloy with some suggestion of an influence on Cb and Ta, the tensile results do not indicate embrittlement of the titanium alloys. They do, however, show definite reduction of ductility for Mo and 302 stainless steel (the bend results for Mo are inconclusive and 302 stainless appears unaffected).

TABLE V

TENSILE TEST DATA
STRAIN RATE 0.01 INCH PER MINUTE

Alloy	Yield Strength (0.2% Offset), ksi		Ultimate Strength, ksi		Per Cent Elongation	
	Wet	Dry	Wet	Dry	Wet	Dry
Titanium	53.5	50	59	68	30	39
Ti-6Al-4V	140.8	136.4	147	157	13	13
Nickel	92.5	89	93	95	8	8
Ni Span C	137	134	143	145	4	5
70/30 Brass	16.7	15.5	47	49	55	65
Mo	64	69.5	74	84	25	35
Cb	50.8	57	68	63	13	12
Ta	77.5	76.4	77.5	78	7.4	5
W	76	72.8	93	76	8	7
Kovar	64	62	78	83	25	26
302 Stainless	68.7	64.7	100	108	57	49
430 Stainless	98.7	96	104	104	8.7	8.3

TABLE VI

TENSILE TEST DATA
STRAIN RATE 0. 10 INCH PER MINUTE

Alloy	Yield Strength (0. 2% Offset), ksi		Ultimate Strength, ksi		Per Cent Elongation	
	Wet	Dry	Wet	Dry	Wet	Dry
Titanium	55. 5	--	68. 8	--	30	--
Ti-6Al-4V	138	--	148. 3	--	10. 2	--
Nickel	86	--	90	--	8. 6	--
Ni Span C	*	--	*	--	*	--
70/30 Brass	15. 8	--	47. 3	--	60	--
Mo	82	--	83	--	43	--
Cb	58	--	68	--	17	--
Ta	59	--	60	--	8. 7	--
W	80. 5	--	80. 9	--	7	--
Kovar	61	--	80	--	27	--
302 Stainless	69	--	102	--	45	--
430 Stainless	95	--	103. 2	--	9	--

* Defective samples.

TABLE VII

TENSILE TEST DATA
STRAIN RATE 0.5 INCH PER MINUTE

Alloy	Yield Strength (0.2% Offset), ksi		Ultimate Strength, ksi		Per Cent Elongation	
	Wet	Dry	Wet	Dry	Wet	Dry
Titanium	62	66	70	74	22	20
Ti-6Al-4V	145	156	151	161	11	12
Nickel	92	94	94.5	96	9	9.5
Ni Span C	144	148	147	150	5	5
70/30 Brass	18	18	47.5	49	59	55
Mo	96.5	98	97.5	99	16	35
Cb	56	55	65	62	14	11
Ta	54.5	80.4	56	81	15	6
W	83	88	86	88	7	8
Kovar	73	69.5	102	86	37.4	7
302 Stainless	66.5	75	80	105	27	35
430 Stainless	102	102	108.5	108.5	10	11

Tantalum shows greater ductility cesium-covered than dry at both extremes of strain rate and definitely lower wetted strength. In all cases the strengths are higher, and the ductilies are lower than one might expect of pure tantalum. The purity and hence the properties are thus suspect, and this result could be specious. Because ductile tensile failures are known to proceed from the inside of a sample outwards, it is difficult to be believe that a surface-active medium could increase the ductility observed. It is, of course, entirely possible for this to happen in a bend or creep test, and rather marked improvements in fatigue life have been observed in the presence of liquid metals[3] due, presumably to a dissolving effect of the liquid either removing the damaged metal or blunting the cracks which form. These particular runs will be repeated.

Comparisons of the stress-strain curves, made with the aforementioned reservations, indicate that the behavior in cesium of some of these materials might not be of the simple type shown on Figure 3. In the case of 302 stainless steel, a definite decrease in the yield strength is seen to occur. This is in contrast to the behavior which is regarded[3] to be general--that is, that the stress-strain curves of susceptible metals are identical, wetted and unwetted, as far as they go. The stress-strain curves for Mo and 302 stainless are shown in Figures 18 and 19.

The wetted Mo (Fig. 18) probably began to crack at the point indicated at A. Up until this point the wetted and unwetted stress-strain curves are virtually identical. For the 302 stainless (Fig. 19), the curves are very different as was mentioned.

In all cases in which embrittlement fails to appear for a couple which is apparently embrittled in another determination, the possibility always exists that wetting is not achieved. For this reason all couples which show embrittlement in any of the tests are suspect and will, of course, be examined in additional detail.

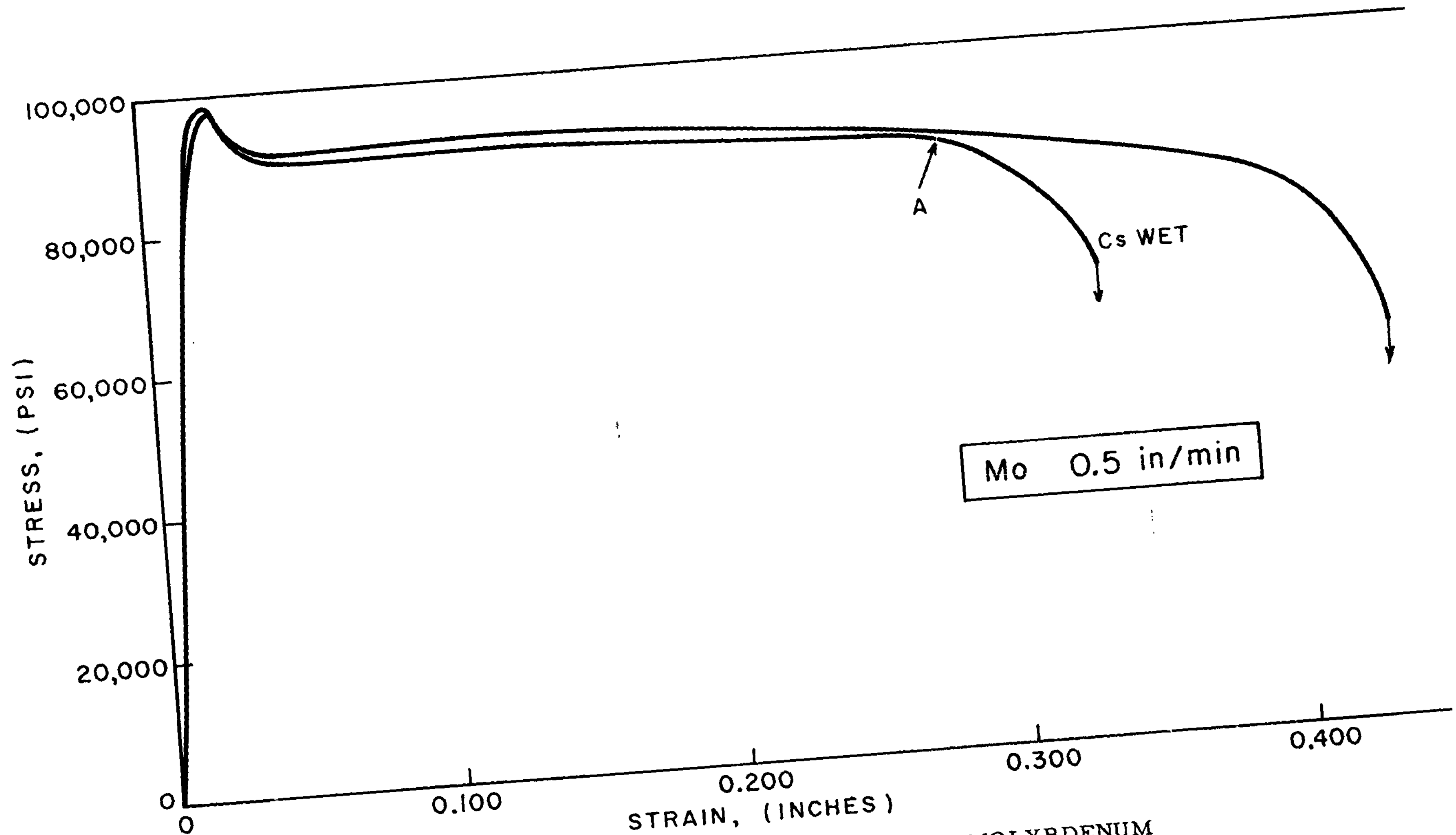

FIG. 18 - STRESS-STRAIN CURVES FOR MOLYBDENUM

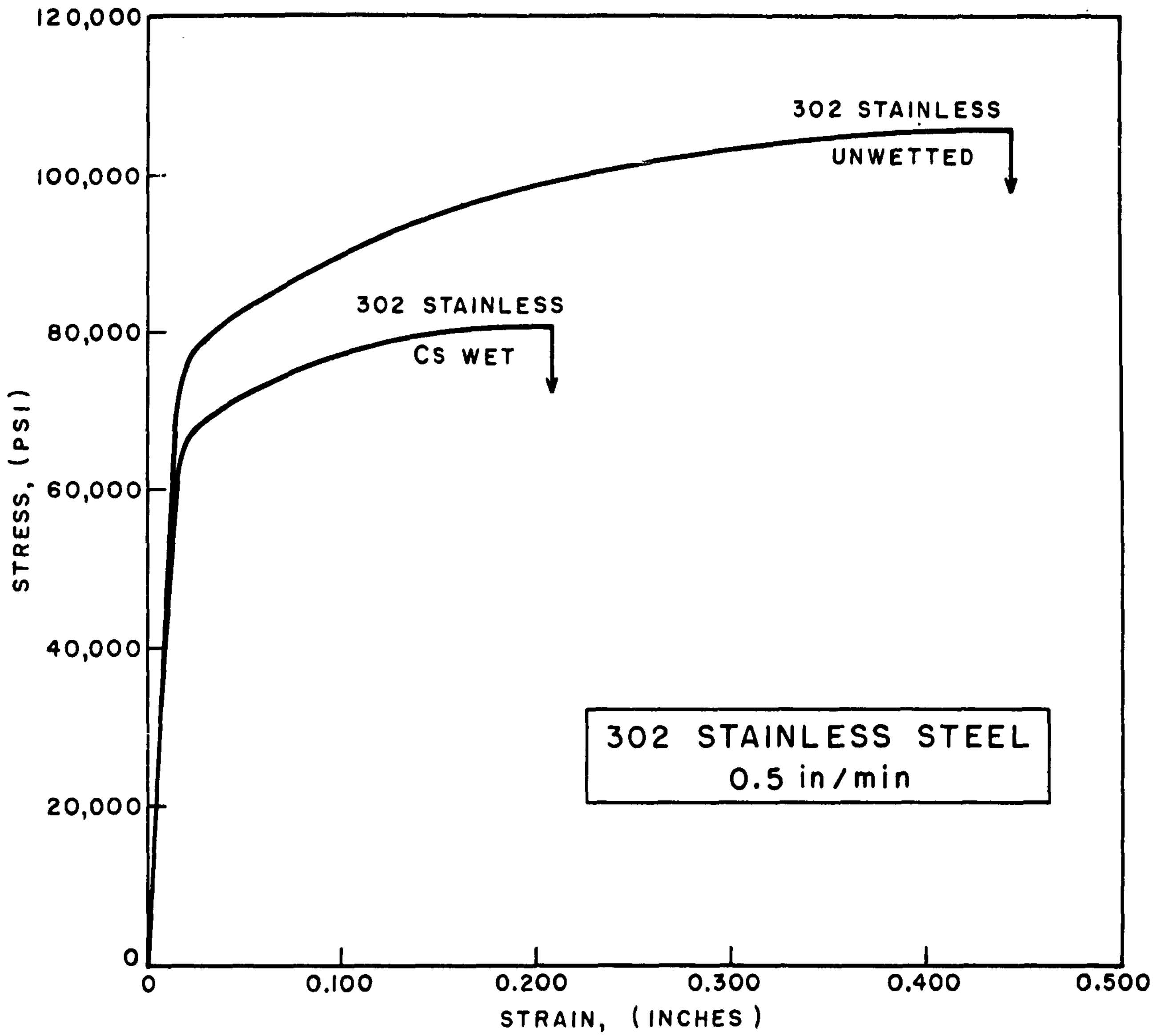

FIG. 19 - STRESS-STRAIN CURVES FOR 302 STAINLESS

Tensile properties for the first of the alumina tied beam tensile samples were gathered and appear in Table VIII. The area in tension was 0.0234 in.^{2} (square section 0.153 in. on a side). These samples are comparable in value with pull-type tensile samples prepared identically and fired with the tied beam specimens. The spread in the data for the pull-type samples is much larger, however, than for the tied beam samples. This, of course, is the principal objection to the use of a simple tensile sample for the ceramic materials.

All of the samples showed fracture in the gage sections perpendicular to the section axis.

E. Other Tests

Some tensile test experiments were carried out using mercury and 70/30 brass, a system of known and high susceptibility. This was done to check out the experimental apparatus and to clarify the effect of both prestrain and strain during the onset of fracture. It was found that for annealed material with a yield stress of 15.5 ksi the wetted fracture occurred at 19.5 ksi. At 19.5 ksi the total plastic strain was 0.023. Additional samples loaded dry to 36 ksi, relaxed, wetted, and reloaded showed no failure at 30 ksi and did not show any sensible plastic flow over the 5-minute period of observation. This sample failed upon loading past 36 ksi. An identical experiment in which the prestrain preload was 40 ksi showed no failure after wetting and reloading until after several seconds had elapsed when 40 ksi was reached and visible plastic flow occurred. The plastic strain required apparently decreases to a very small value as the yield strength rises.

This result is consistent with the delayed failure result previously reported. Both clearly show that plastic flow is necessary for the production of mercury induced brittle fracture in 70/30 brass and implies strongly that the delayed failure phenomenon must await the necessary strain by a creep mechanism. It is not yet clear whether this is a crack nucleation requirement or a prerequisite for propagation of an already existing crack to a stable size.

TABLE VIII

TENSILE STRENGTH OF WESGO Al-995

Specimen	No. of Tests	Fracture Strength, psi	Min., psi	Max., psi	2nd Min., psi	2nd Max., psi
Pull type	5	14,669	12,096	18,434	12,873	16,949
5 in. tied beam	9	13,257	10,939	14,154	12,213	14,036

TABLE IX

SUMMARY OF EFFECTS OBSERVED

Alloy	Yield Strength Reduced	Ultimate Tensile Strength Reduced	% Elongation Reduced	Other
Titanium	--	X	--	FDB
Ti-6Al-4V	?	X	--	FDB
Nickel	--	--	--	Not affected
Ni-Span-C	--	--	--	Not affected
70/30 Brass	--	--	--	Not affected
Mo	--	--	X	--
Cb	--	--	--	CDB
Ta	?	?	--	CDB
W	--	--	--	Not affected
Kovar	--	--	--	Not affected
302 Stainless	X	X	X	High strain rate only
430 Stainless	--	--	--	Not affected
BT Solder	--	--	--	FDB
B120 VCA	--	--	--	CDB

FDB = fails dynamic bend wetter

CDB = small cracks in wetted dynamic bend

VI. SUMMARY

The results obtained to date on the effect of molten cesium on the mechanical properties of some of the materials found in thermionic energy converters are summarized in Table IX. In all cases so far examined there is no evidence of catastrophic embrittlement. Whether this will remain true as higher strength conditions are studied remains to be seen. It is expected that some increase in the severity of the embrittlement will be observed under conditions where the cesium is molten and the temperature is low. It is, however, also expected that this will be offset to some degree when the cesium is in the gas phase rather than liquid and offset entirely above the transition temperatures.

It is the intent during the forthcoming period to expand the materials examined to include mild, low alloy and PH steels, additional nickel-base alloys, and alloys based upon refractory metals, and high-alumina ceramics.

Those materials which appear to be affected will be examined in far greater detail with cesium in both the liquid and gaseous state.

VII. PERSONNEL AND LOGBOOKS

The work reported herein was performed by D. W. Levinson, K. L. Coleman, and S. A. Bortz. Original data may be found in ARF Logbooks Nos. C-12776 and C-12687.

Respectfully submitted,

ARMOUR RESEARCH FOUNDATION OF
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Definite reductions in tensile ductility were observed for 302 stainless steel and molybdenum. Bend ductility was lowered in the cases of unalloyed titanium, titanium -6 aluminum -4 vanadium alloy, columbium, tantalum and silver-copper eutectic solder.

Some difficulty in reproducibly wetting samples was evident throughout the study. Slightly contaminating the cesium with oxygen (or water) was observed to increase cesium wettability markedly.

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